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# Wear Testing of Zirconia Coatings for Applications in Total Hip Replacement

Carrie Stewart, Alan Eberhardt

## Abstract:

*Polyethylene wear is recognized as one of the leading contributors in the long-term failure of total hip replacements (THR). Improving the tribological properties of the articulating surfaces in THR reduces wear and extends implant life. Zirconia is a biocompatible material that has been used in femoral heads because of its high hardness and smooth finish. Presently, twelve different metallic-coating combinations were tested, including anodized and non-anodized commercially pure titanium (CP-Ti) and titanium alloy (Ti6Al4V), each with one, two or no coats of zirconia. Zirconia-coated CP-Ti and Ti6Al4V were wear-tested against ultra high molecular weight polyethylene (UHMWPE) as a preliminary screening of the coatings as candidates for use in THR. CP-Ti coated with one zirconia layer was found to cause less UHMWPE mass loss than the uncoated alloys and twice coated alloys. The remaining coated samples, however, showed more mass loss than the uncoated alloys. Meta-analysis suggested the thicknesses for the twice coated specimens may have exceeded the critical thickness values for zirconia coatings, leading to the poor performance in the present comparisons.*

## Introduction

Ultra high molecular weight polyethylene (UHMWPE), the most common articulating surface used in the acetabular component of total hip replacements (THR), exhibits low friction and wear behavior; however, there remain concerns regarding its longterm performance. In the 1990s, UHMWPE wear debris became recognized as one of the central causes for initiating osteolysis, a major factor that limits joint prosthesis life.[1] Particulate wear debris circulates in body-fluids around the total joint prosthesis and initiate a foreign body reaction in local bone. Studies suggest that over $10^5$ polyethylene particles are formed with each step.[2] Cells responsible for destroying foreign matter are activated by the polyethylene particles, and this process simultaneously destroys peri-implant tissues, including bone, through osteolysis.[2]

Titanium alloys are suitable materials for total hip replacement because they are very resistant to corrosion, which is a good indicator of biocompatibility. An oxide film, $TiO_2$, passively forms and protects both commercially pure titanium (CP-Ti) and its alloy (Ti6Al4V).[5] A drawback to titanium and its alloy is the relative softness as compared to other metals. Titanium alloys are not commonly used for femoral heads in total hip replacement because they perform poorly in comparison to the cobalt-chromium (CoCr) bearing surfaces currently popular in THR.

Modern THRs are typically modular and allow different material combinations of metals, ceramics and polyethylene components, which affect tribological behaviors. The CoCr and UHMWPE combination has been the standard for THR since the 1970s. Metal-on-metal implants were one of the earliest successful combinations, and newer, alternative, biocompatible bearing surfaces have the potential to increase the lifespan of a THR.[4] Increased implant life would be especially beneficial to younger patients and patients with high activity levels. Material choices in THR are limited by manufacturing restrictions and precise implantation techniques.[5]

Many bearing surfaces have been coated with different materials to manipulate tribological behavior. Alumina, zirconia, nitriding, and nitrogen ion implantation of Ti alloys [7], and amorphous diamond have all been used to try to improve tribological properties in THR. Alumina, with high hardness, fracture toughness, and biocompatibility, has been clinically used as a bearing surface in alumina-UHMWPE configurations.[8,17] Wear rates of zirconia ceramics, yttria-stabilized tetragonal zirconia polycrystal (Y-TZP), have shown lower wear rates when articulated against UHMWPE as compared to alumina.[6]

The purpose of this study was to screen zirconia surface coatings on Ti6Al4V and CP-Ti for potential bearing surfaces of femoral heads against UHMWPE in THR. It was hypothesized that of the materials tested, zirconia coated Ti6Al4V and CP-Ti would outperform the uncoated titanium surfaces. In other words, the hard, smooth coating of zirconia on the metallic was expected to cause less UHMWPE wear.

## Materials and Methods

### *Materials*

CP-Ti and Ti6Al4V alloy samples were provided by an external university source. Coatings were applied to the samples by the source using proprietary sol-gel spin coating methods. There were three or four samples of each specific metallic and coating combination. Table 1 illustrates the testing matrix for the different alloys.

UHMWPE (Arcom®, Biomet, Inc., Warsaw, IN) was machined into cylindrical wear pins 9.5 mm in diameter with a step down to 4.76 +/- 0.03 mm diameter on the contacting surface, giving a contact area of approximately 17.8 $mm^2$. The pins were soaked in bovine serum (Hyclone, Logan, UT) for a minimum of two weeks prior to wear testing.

### 1. *Frictional Wear Testing*

Before tests began, pins and disks were sonicated in a 10% LiquiNox® (Alconox Inc., White Plains, NY) solution, rinsed with deionized water, and submerged in ethyl alcohol. Then the pins and disks were dried in a vacuum for thirty minutes. To obtain a change in mass, the initial and final pin weights were recorded. The pins were each weighed five times on a Mettler Toledo AG245 microbalance (Columbus, OH) with a resolution to .00001 grams and averaged, before and after wear testing.

During each test, the pins and disk samples were submerged in a lubricant that contained 53% bovine serum water, anhydrous ethylenediaminetetraacetic acid (EDTA) and sodium azide. EDTA (Sigma-Aldrich, St. Louis, MO), was added to bind the calcium in the solution and prevent precipitation of abrasive components onto the bearing surfaces. Sodium azide was also added to prevent bacterial depredation. The mixture was filtered with a .22 micron filter, and warmed to 37° C. The lubricant temperature was maintained at body temperature throughout testing.

Wear testing was performed in an OrthoPOD® six station pin-on-disk machine (Advanced Mechanical Testing, Inc., Watertown, MA). Nine tests were performed. The CP-Ti samples were tested separately from the Ti6Al4V samples. Each test, of the same metallic type, had a coated or uncoated sample at five of the six stations. A mixture of coating types was used during the test. Six pins and five disks were used during each test. The sixth pin was used for as an unworn soak control.

The polyethylene pins were installed into the OrthoPOD® and articulated against the flat, coated test faces of the metal disks. Figure 1 shows the configuration of the pin and disk. The wear path followed a figure eight pattern at a frequency of 1.5 Hz and a sliding speed of .4 m/s at constant load of 10.7 MPa. Testing conditions were chosen in accordance with ASTM standards G99 and F732. The OrthoPOD® software calculated coefficient of friction based on three triaxial load cells at intervals of 25,000 cycles. The horizontal force measurements had an accuracy of +/- 2N, and the vertical forces were accurate +/- 8N. Each test length was 500,000 cycles.

After 500,000 cycles, the pins and disks were removed from the machine, soaked for two days, mass was measured again, and mass loss was calculated. The soak control accounted for any change in mass due to fluid absorption.

### *Statistical Analysis*

Kruskal-Wallis one-way analysis of variance by ranks was applied to test if there were overall differences in mass loss among the different test groups. This form of non-parametric statistics substitutes the ranking of the wear value for the actual value and the sum of ranks is used to test for differences among the groups. Additional t-tests were performed to compare different treatments, with a level of statistical significance of $\alpha = 0.05$. StatView software was used to perform both the non-parametric and parametric tests. The treatments compared were anodized and non-anodized samples as well as coated and uncoated samples. Samples were randomly selected for visual observation of wear paths using light microscopy (KEYENCE, Osaka, Japan).

## Results

The four best performing surfaces were CP-Ti once coated with $ZrO_2$, uncoated anodized CP-Ti, uncoated Ti6Al4V, and uncoated anodized Ti6Al4 with average mass loss for the surfaces ranging from 660 μg to 933 μg. The four worst performing surfaces were all twice coated with $ZrO_2$, with average mass loss for the surfaces ranging from 51.5 mg to 7.5 mg. Uncoated CP-Ti, anodized CP-Ti once coated with $ZrO_2$, Ti6Al4V once coated with $ZrO_2$, and anodized Ti6Al4V once coated with $ZrO_2$ coated were ranked among the middle of the pack. Figure 2 illustrates the mass loss associated with each coating.

The Kruskal-Wallis analysis indicated that there were statistically different wear rates among the groups ($p < 0.001$), and provided a ranking of best to worst. The mean mass losses associated with the coated specimens was 57% higher than the uncoated specimens; however the differences were not significant (p=.0634). Among the anodized and non-anodized samples, mean mass losses were 54% higher among the anodized; however, again these results were not significant (p=.0687).

Evidence of material transfer was apparent along the wear path on the coated specimens following wear testing. Figure 3 shows a magnified image confirming material transfer onto the surface of the disk. The transfer film was likely UHMWPE, since UHMWPE has been known to transfer to counter-surfaces in wear testing.[16] Due to the proprietary nature of the coating processes, all specimens were returned to the manufacturer after wear testing was completed, and no further analyses were performed.

## Discussion

The original hypothesis, that the $ZrO_2$ coatings on the titanium surfaces would create less wear than the uncoated titanium surfaces was supported in the case of the once coated CP-Ti. The next best performing surfaces were all uncoated. The remaining coated surfaces exhibited poorer wear behavior than the once coated CP-Ti. CP-Ti may have outperformed Ti6Al4V because of the latter material's surface instability. Ti6Al4V surface breakdown has been reported as one of the main causes of poor polyethylene wear performance. Delamination of the titanium alloy's surface layer has been reported to increase polyethylene wear. Polymer binding increases surface roughness and has been associated with the poor wear performance exhibited by Ti6Al4V. [5]

The history of zirconia in femoral heads reveals mixed results with regard to polyethylene wear due to the complex nature of the material. Each zirconia implant has unique properties specific to the manufacturer, and therefore not all zirconia coatings have the same tribological properties. Y-TZP has high fracture toughness, as compared to other ceramics like alumina, which has been equated to better reliability. Also, a Y-TZP has a lower Young's modulus and ability to be polished to a superior surface finish as compared to other ceramics [11,3] Y-TZP undergoes transformation toughening on machined and polished bearing surfaces, whereby three to five percent transforms from tetragonal phase to the monoclinic phase. The monoclinic phase change results in small volume increases and there is a volumetric expansion of the ceramic grains that cause a compressive surface layer that helps to resist crack initiation and propagation. [10]

While transformation toughening is said to account for the high strength of the material, under certain manufacturing conditions of stress and moisture, the Y-TZP can transform more aggressively to the monoclinic phase than is desired. Increased monoclinic phase in zirconia increases the surface roughness and has shown poor performance and wear results when articulating with polyethylene.[10] Additionally, several studies have reported difficulties in obtaining crack-free coatings with variable thicknesses of zirconia.[12]

Recent studies have shown that ceramics can have properties such as decreased surface roughness, increased hardness and scratch resistance, which are a benefits over metallics. The benefits have shown clinical significance in improving wear behavior of articulating surfaces. [10,3] In the present study, the zirconia coatings tested may have been thicker than the critical thickness for zirconia coatings, which the manufacturer of the samples reported as 200nm.[9] Critical thickness measurements are based on the internal stresses of the zirconia and are related to the sol-gel deposition methods.[15] No additional information was provided by the manufacturer, and no analysis of coating thickness was conducted in the present study.

One potential benefit of using ceramics over metallics in implants is the possibility of obtaining superior surface finishes. However, in this experiment, only the single coated CP-Ti outperformed the uncoated samples. The majority of surface treatments of the metallics in this study did not display advantages in wear performance against polyethylene as compared to single coated CP-Ti. The titanium alloys coated twice with zirconia performed poorly, among the worst of the samples, suggesting that the added coating thickness was not advantageous to the tribological properties of the titanium and ceramic combination. The manufacturer provided no information regarding the polishing or final surface roughness values.

Future studies dealing with tribological properties of titanium metallics coated with zirconia ceramics should concentrate on applying crack-free coatings by obtaining a coating on the alloy less than the critical thickness. The thicknesses at which coatings are crack-free vary based upon application processes, materials, and methods used to apply the ceramics to alloys. [12,13] The applied thickness of coatings should be slightly less than the critical thickness to prevent crack formation.[14] Optimizing the coating fabrication process will be important for the future of ceramic coatings on titanium alloys for hip implants.

## Conclusions

In summary, a series of coated and uncoated specimens were wear tested against UHMWPE to compare the behaviors of two alloy substrates (CP-Ti and Ti6Al4V), under anodized and non-anodized conditions, and with one or two layers of zirconia coating. It was hypothesized that the zirconia coated specimens would outperform the uncoated Ti-based substrates. The results indicated that the twice coated specimens were associated with mass losses that were an order of magnitude greater than the non-coated or once coated specimens. No significant differences in mass lost were detected when comparing the base substrates, nor between the anodized and non-anodized specimens. Meta-analysis suggested that coating thicknesses exceeding critical thickness may have been linked to the poor wear performance of the twice-coated zirconia specimens.

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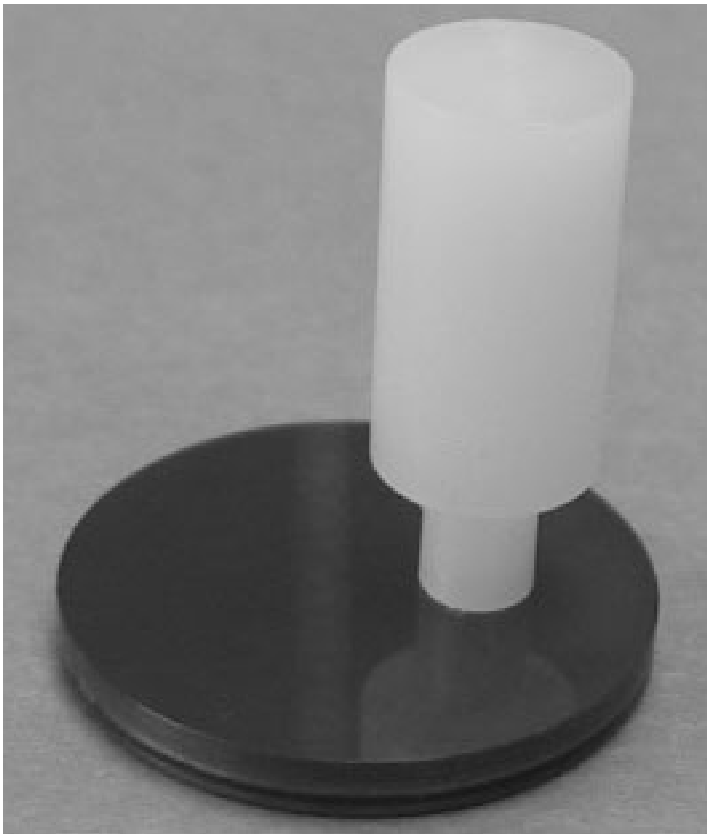
Table 1

| **Base Material** | **Treatment** |
|---|---|
| CP-Ti<br>N=4 | No coating<br>$ZrO_2$ Coated at 550° C<br>$ZrO_2$ Coated at 550° C, 2 x $ZrO_2$ Coated layers |
| 50V Anodized<br>Cp-Ti<br>N=4 | No Coating<br>$ZrO_2$ Coated – 550° C<br>Anodized, $ZrO_2$ Coated at 550° C, 2 x $ZrO_2$ Coated layers |
| Ti6Al4V<br>N=3 | No Coating<br>Ti6Al4V - $ZrO_2$ Coated at 550° C<br>Ti6Al4V - $ZrO_2$ Coated at 550° C, 2 x $ZrO_2$ Coated layers |
| 50V Anodized<br>Ti6Al4V<br>N=3 | No Coating<br>$ZrO_2$ Coated at 550° C<br>$ZrO_2$ Coated at 550° C, 2 x $ZrO_2$ Coated layers |

Figure 1. The pin and disk configuration for articulation.



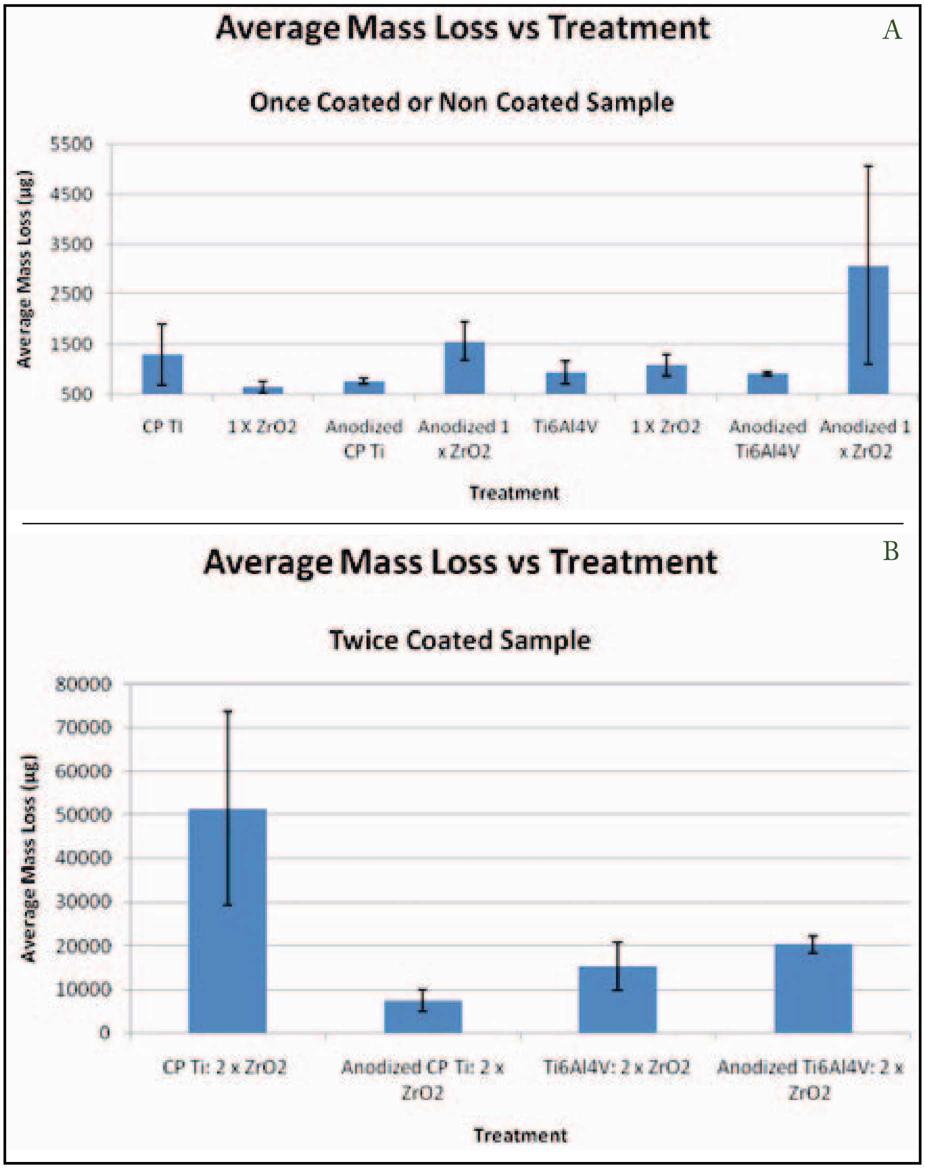


Figure 2. (a) Mean mass loss (bars indicate standard deviation) for each of the 8 metallic and once-coated combinations used as counter faces during wear testing; (b) Mean mass loss for the twice zirconia-coated specimens illustrating values one order of magnitude higher than the non- or once-coated specimens.

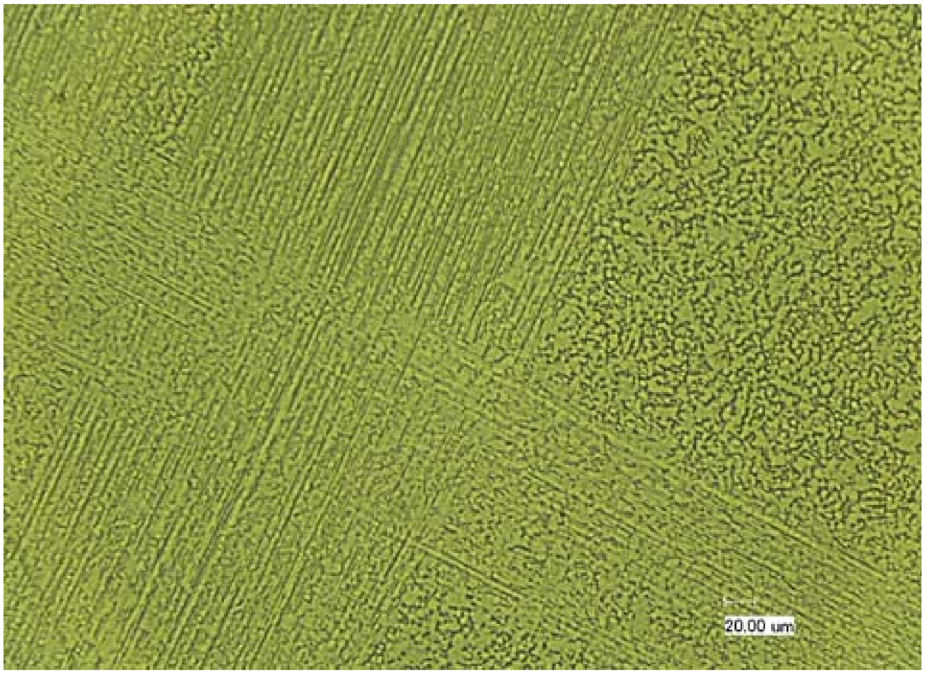


Figure 3. Keyence micrograph reveals evidence of a transfer film at the crossing point of the Figure-8 wear path between the UHMWPE pin and twice-coated disk.